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Spectrometric investigation of complexes obtained by interaction of tin(II) and platinum(IV)

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The formation of three forms of Pt(II)–Sn(II) complexes during a reaction of Pt(IV) and Sn(II) solutions, depending on the HCl, $SnCl_2$ and total Cl^- concentrations, has been shown by means of light absorption and X-ray photoelectron spectroscopy. The transition of $H_3[Pt(SnCl_3)_5]$ complex to $H_2[Cl_2Pt(SnCl_3)_2]$, and of $H_2[Cl_2Pt(SnCl_3)_2]$ complex to $H_2[Pt_3(SnCl_3)_8]$, observed over time, is related to the oxidation of Sn(II). An increase in temperature accelerates the transition. The initial complexes may be renewed by supplementing the missing amounts of $SnCl_2$ and HCl. An increase in the $SnCl_2$ concentrations stabilizes the complexes.

Key words: platinum(IV), tin(II), Pt(II)-Sn(II) complexes, light adsorption

INTRODUCTION

The platinum complex and colloid solutions prepared in different methods have particular importance in the preparation of activators and catalysts for various applications [1, 2]. The catalytic activity of Pt nanoparticles and clusters varies in the method of their preparation – radiolysis or reduction with H_2, citrate, $NaBH_4$ – even if the same stabilizes are used [3]. These distinctions may be conditioned by different sizes and shapes of the particles prepared. The stabilizers used in the solutions block the activity of Pt colloid. Platinum nanoparticles are also used for the modification of carbon, silicon, TiO_2, organic compounds and plastics surface [4–7]. The immobilized Pt colloids were found to be active and stable heterogeneous catalysts. The results of the $SnCl_2$ reaction with several organometalic substrates of platinum have been reported, where $SnCl_2$ has been inserted into Pt–Cl bounds [8].

The reaction of platinum with tin(II) has been used for a long time for colorimetric determination of small amounts of platinum [9]. The composition of the products of the interaction of platinum with $SnCl_2$ is not entirely clear, since the data and opinions of authors are contradictory. Some authors [10, 11] observed the formation of a brown product after tin(II) was added to a platinum chloride solution. This product was attributed to colloids because it did not pass through semi-permeable membranes. Other authors [12–15] described a rapid formation of a stable yellow to red solution suitable for colorimetric analysis. This compound has been attributed to a platinum(II) or chloroplatinous acid complex with tin(II). Extensive studies of the reaction between Pt(II) and Sn(II) chlorides in a hydrochloric acid solution performed by the spectrometric method indicated the existence of several reaction products. In an aqueous medium, the principal product corresponded to the tin–platinum ratio 5 : 1 [16, 17]. However, the opinions concerning the state of platinum in the complexes differed. According to some authors [12–14], one mole of tin(II) in the reaction was oxidized to tin(IV) simultaneously with the reduction of Pt(II) to Pt(0). This opinion was confirmed by means of potentiometry and polarography [13]. The intensive color of solution was attributed not to a platinum colloid, but to a complex of $SnCl_2$ with platinum in zerovalent state. It has been found by the spectrometric method that in solutions, containing 0.5–2 M of chlorides, platinum forms two complexes with stannous chloride [14]. The existence of *ciss-trans* isomeric forms of the platinum complex with $SnCl_2$ was suggested.

Many authors tried to examine the products formed from $SnCl_2$ and $PtCl_2$ by precipitation in various solvents. The salts of $[Cl_2Pt(SnCl_3)_2]^{-2}$ and $[Pt(SnCl_3)_5]^{-3}$ anions were precipitated with organic compounds [16, 17]. By means of nuclear gamma resonance spectra, it was found that tin presenting in the complexes is not oxidized to IV and is bound by a covalent bond with platinum(II), but not by the ionic one [16]. An investigation of the products formed in an acetone solution showed the existence of a third anionic complex, $[Pt_3Sn_8Cl_{20}]^{-4}$ [15]. What makes this form interesting is that the platinum atoms are apparently reduced to the zerovalent state. It has been determined by means of gamma resonance, rentgenoelectrone and light absorption spectra that in the solutions with the ratio Cl : Pt ≥ 1000 the compounds of Pt(II) are formed [18]. When the ratio of Cl : Pt ≤ 300 and Sn(II) : Pt(II) ≤ 5, the complexes of Pt–Sn with a lower degree of platinum oxidation were detected. At the ratio Cl : Pt ≤ 6 and Sn(II) : Pt(II) = 1, platinum was reduced by tin(II) to metal. The complexes $(Me_4N)_3[Pt(SnCl_3)_5]$, $(Me_4N)_2[Pt(SnCl_3)_2Cl_2]$,

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$(Me_4N)_6[Pt_3(SnCl_3)_8]$, $(Me_4N)_4[Pt_3(SnCl_3)_5]$ were separated (Me means methyl $-CH_3$). By the reaction of $[PtCl_4]^{2-}$ with $SnCl_2$, depending on the molar ratio, several derivatives $[PtCl_n(SnCl_3)_{4-n}]^{2-}$ were obtained; even $Pt(SnCl_3)_5]^{3-}$ can be isolated by using an excess of $SnCl_2$ [19], although the instability of the Pt–Sn bonds very often leads to mixtures of different species in equilibrium [20].

Data of different authors often disagree probably because of different conditions and concentrations of the reactants used. The aim of this study was to estimate the differences between the products formed in the reaction of low Pt(IV) concentrations with an excess of $SnCl_2$ in hydrochloric acid solutions and to investigate their stability and change over time.

EXPERIMENTAL

Platinum–tin derivatives were prepared using two main stock solutions: 5 mM H_2PtCl_6 and 50 mM $SnCl_2$. The Pt(IV) solution was prepared by dissolving $H_2PtCl_6 \cdot 6H_2O$ in water. The Sn(II) solution was prepared by dissolving $SnCl_2$ in concentrated HCl under heating and then diluting with water. Freshly prepared tin salt solutions were always used to minimize the effect of tin oxidation by the atmospheric oxygen before the reaction with Pt(IV). Analytical grade chemicals and triply distilled water were used to prepare the solutions. The Pt(IV) and Sn(II) solutions, diluted to the concentrations needed, were mixed by adding tin salt to platinum(IV) after adjusting pH of the solutions. The platinum concentration in solutions was 0.1–0.5 mM.

The complexes obtained were characterized by light absorption spectra using a Perkin-Elmer Lambda 35 UV / Vis spectrometer. The solutions were diluted fivefold with HCl (1 : 10) prior to each spectrometric measurement. It was established that such dilution did not change the shape of spectrum. Water was used as the reference solution.

The derivatives formed in the solutions were studied by electron microscopy using an electron microscope TEM MORGAGNI 268. The platinum–tin solutions for investigations of the formed nanoparticles were dropped on a copper net preliminary covered by a carbon film.

Elemental analysis of platinum–tin complexes and the valence state of platinum and other elements was performed using the XPS method. Plates of glass or glassy carbon were immersed into the solutions of platinum–tin complexes for 30 minutes and then dried. Spectra were recorded with a Vacuum Generator (VG) ESCALAB MK II spectrometer. The non-monochromatic Al Kα X-ray radiation ($h\nu$ = 1486.6 eV) was used for excitation, and the Al twin anode was operated at 14 kV and 20 mA. The photoelectron take-off angle was 45° with respect to the normal sample surface, and the spectra of Pt4f, Sn3d, O1s, Cl2p, C1s and Si2p were taken at the constant analyzer energy mode (20 eV pass energy). The base pressure was kept at below 5×10^{-8} Torr. The spectrometer was calibrated relative to Ag3d5/2 at 368.0 ± 0.1 eV and Au4f7/2 at 83.8 ± 0.1 eV.

RESULTS AND DISCUSSION

The stability of platinum derivatives obtained by reducing 0.1–0.5 mM H_2PtCl_6 solutions with Sn(II) in the hydrochloric acid medium depends on the initial $SnCl_2$ and HCl concentrations. When the Sn(II):Pt(IV) ratio is lower than 5 there forms a brown turbid and unstable solution is formed, that precipitates in a few hours. The Pt–Sn derivative formed with the increased initial Sn(II) concentration is relatively stable. However, the complex obtained at HCl concentration lower then 10 mM is unstable as well. Therefore, stable Pt–Sn derivatives may be formed only at certain initial concentrations of the reactants.

The light absorption spectra of the yellow Pt–Sn compound, obtained at HCl concentration over 1 M (Fig. 1a), show that the height of the maximums at 310 and 400 nm is directly proportional to the initial Pt(IV) concentration, therefore, this compound is suitable for Pt colorimetric estimation. This compound was attributed to a Pt(0)–Sn(II) complex *ciss* form [14], and / or to the anion $[Pt(SnCl_3)_5]^{3-}$, in which platinum(II) is five coordinated [16, 17]. In our work, this complex is specified as form I. It changes over time – the optical density at 310 and 410 nm slightly decreased after 25 hours. After two days, the character of the spectra changed (Fig. 1b) as the initial maximums disap-

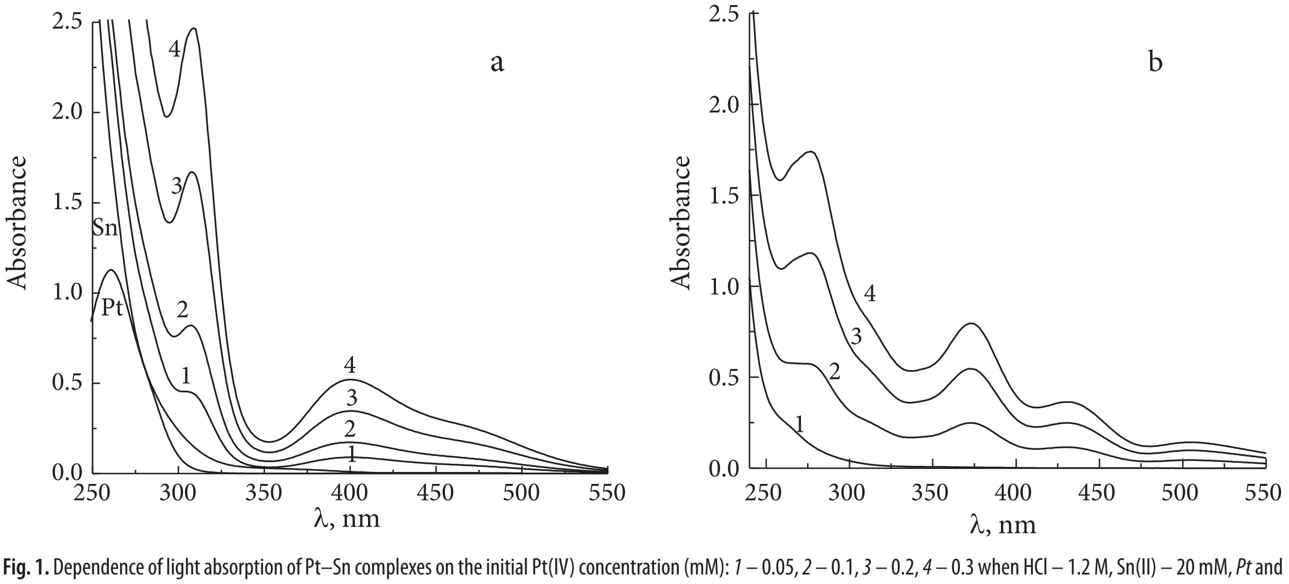

Fig. 1. Dependence of light absorption of Pt–Sn complexes on the initial Pt(IV) concentration (mM): *1* – 0.05, *2* – 0.1, *3* – 0.2, *4* – 0.3 when HCl – 1.2 M, Sn(II) – 20 mM, *Pt* and *Sn* – spectra of platinum (0.3 mM) and Sn(II) recorded under the same conditions separately; *a* – just prepared, *b* – after two days. Solutions diluted 1 : 4 with HCl 1 : 10

peared, and increased absorption at 275, 375, 430 and 510 nm occured (form II). A yellow to red complex, distinguished by these maximums in light absorption spectra, was obtained in the Pt(II) and Sn(II) mixture when the concentration of HCl was 0.5 M and was attributed to a *trans* form of Pt(0)–Sn(II) complex [14]. In accordance to nuclear gamma resonance spectra [16, 17], it may be specified as $[Cl_2Pt(SnCl_3)_2]^{-2}$, in which Pt(II) is four coordinated. Further changes in the obtained derivative over time lead to a degradation and discoloration of the complex, primarily containing the lowest initial Pt(IV) concentration. It is worth noting, that the authors, who proposed Pt–Sn(II) isomeric forms [14], used only freshly prepared complexes and did not observe the transition of the *ciss* form to the *trans* one. They obtained the *ciss* form in 1–2 M HCl solutions and *trans* form – in 0.25–0.5 M HCl solutions.

Even three forms of freshly prepared platinum–tin derivatives may be identified in the light absorption spectra of the solutions containing the initial concentration of $SnCl_2$ 20 mM, and HCl from 0.15 to 1.1 M (Fig. 2a). The formation of form I is clearly seen (maximums at 310 and 400 nm) from the spectra of solutions with the concentration of HCl close to 1 M. Form II may be found from the increased absorption at 370 and 430 nm in the spectrum of the complexes, formed at the concentration of HCl close to 0.5 M (Fig. 2a, curve 2). The spectrum of a brown solution obtained with a low HCl concentration in the mixture (0.25 M) has no maximums and is similar to the spectrum of spherical 10 nm particles of the metallic Pt [21]. It seems, that under these conditions, Pt(IV) might be reduced to Pt(0), and according to [15], could be treated as anion $[Pt_3Sn_8Cl_{20}]^{-4}$; every atom of Pt in this complex requires the lowest concentration of chloride as compared with that of forms I and II. This Pt complex prepared at a low HCl : $SnCl_2$ ratio (form III) becomes turbid after 20 h and precipitates after the next two days. A common tendency is observed – the transition of form I to form II, and form II to form III (Fig. 2b), the latter being most characteristic of colloids. A comparison of the stability of all the complexes produced under the conditions of Fig. 2 suggests that the complexes containing a higher initial $SnCl_2$ concentration are more stable.

The variation in the character of the spectrum of freshly prepared platinum–tin derivatives, depending on the concentration of $SnCl_2$, is distinctly seen in Fig. 3. When 0.5 M HCl and 5–10 mM $SnCl_2$ are used (HCl : $SnCl_2$ = 50–100) the product corresponds to form II (Fig. 3a). With an increase in the initial $SnCl_2$ concentration, the light absorption spectrum changes to form III. The transition to form III is also clearly seen with an increase in time after the preparation of the complexes (Fig. 3b), when the light absorption maximums characteristic of form II reduce gradually. The complex prepared with the lowest $SnCl_2$ (5 mM) concentration precipitates after two days. The deposits may be dissolved by adding HCl. The complex prepared with the highest Sn(II) concentration (50 mM) changes somewhat slowly.

When the initial HCl concentration is higher than 1 M, the light absorption spectrum always shows the formation of complex I, the amount of which, clearly seen from the height of the maximum at 310 nm (Fig. 3c), increases with an increase in the initial $SnCl_2$ concentration. The complex formed at a low $SnCl_2$ concentration (5 mM) is unstable, it fades after two days because of lack of $SnCl_2$ (Fig. 3d). The solution prepared with 50 mM $SnCl_2$ remains stable for a considerably longer time (6–7 days). If the initial $SnCl_2$ concentration in the solutions is lower than 50 mM, the light absorption maximum at 400 nm shifts in the direction of shorter waves, finally testifying the transition of form I to form II. The same transition to form II is observed when heating a freshly prepared complex of form I, in this case, the transition is very rapid – 10–20 min at 80 °C.

Electron microscope images of platinum–tin compounds in Fig. 4 show that the size of nanoparticles becomes smaller with an increase in HCl concentration in the Pt(IV) and Sn(II) mixture. This is conditioned by the complex state in the solution. Particularly fine nanoparticles are formed when the concentration of HCl is 1 M and higher (complex of form I). With the transition of the complex to forms II and III, the nanoparticles become coarser. The sizes of the nanoparticles diminish also with an increase in the $SnCl_2$ concentration in the initial mixture. This is natural because the addition of $SnCl_2$ shifts the reaction to the

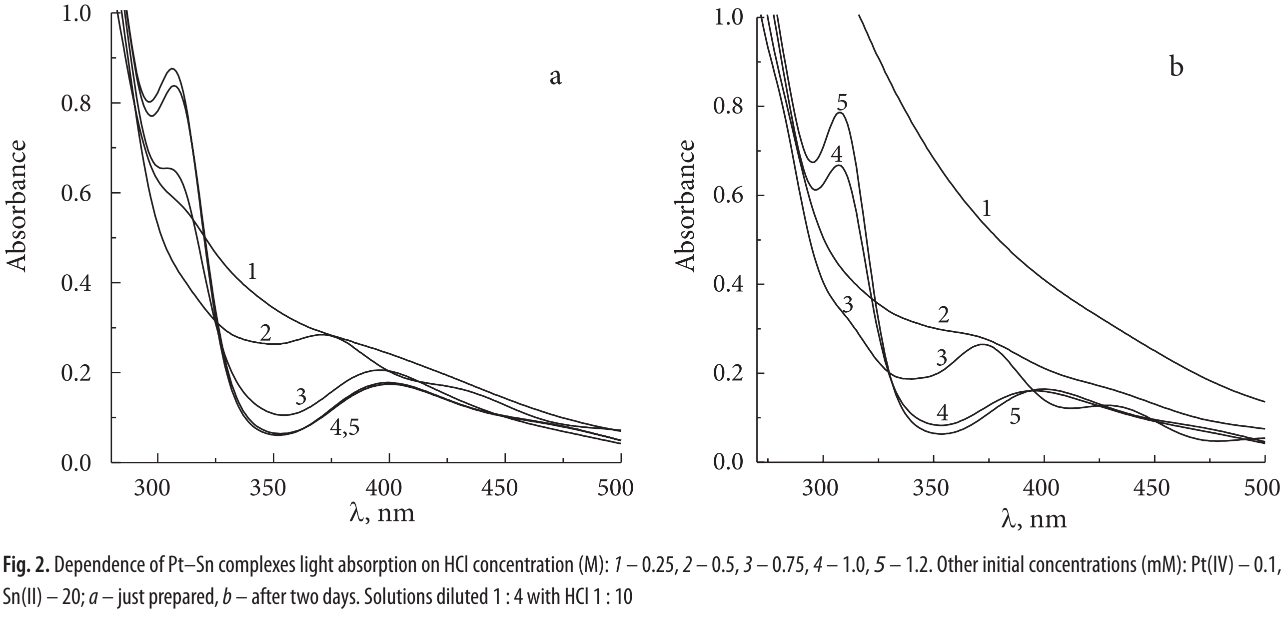

Fig. 2. Dependence of Pt–Sn complexes light absorption on HCl concentration (M): *1* – 0.25, *2* – 0.5, *3* – 0.75, *4* – 1.0, *5* – 1.2. Other initial concentrations (mM): Pt(IV) – 0.1, Sn(II) – 20; *a* – just prepared, *b* – after two days. Solutions diluted 1 : 4 with HCl 1 : 10

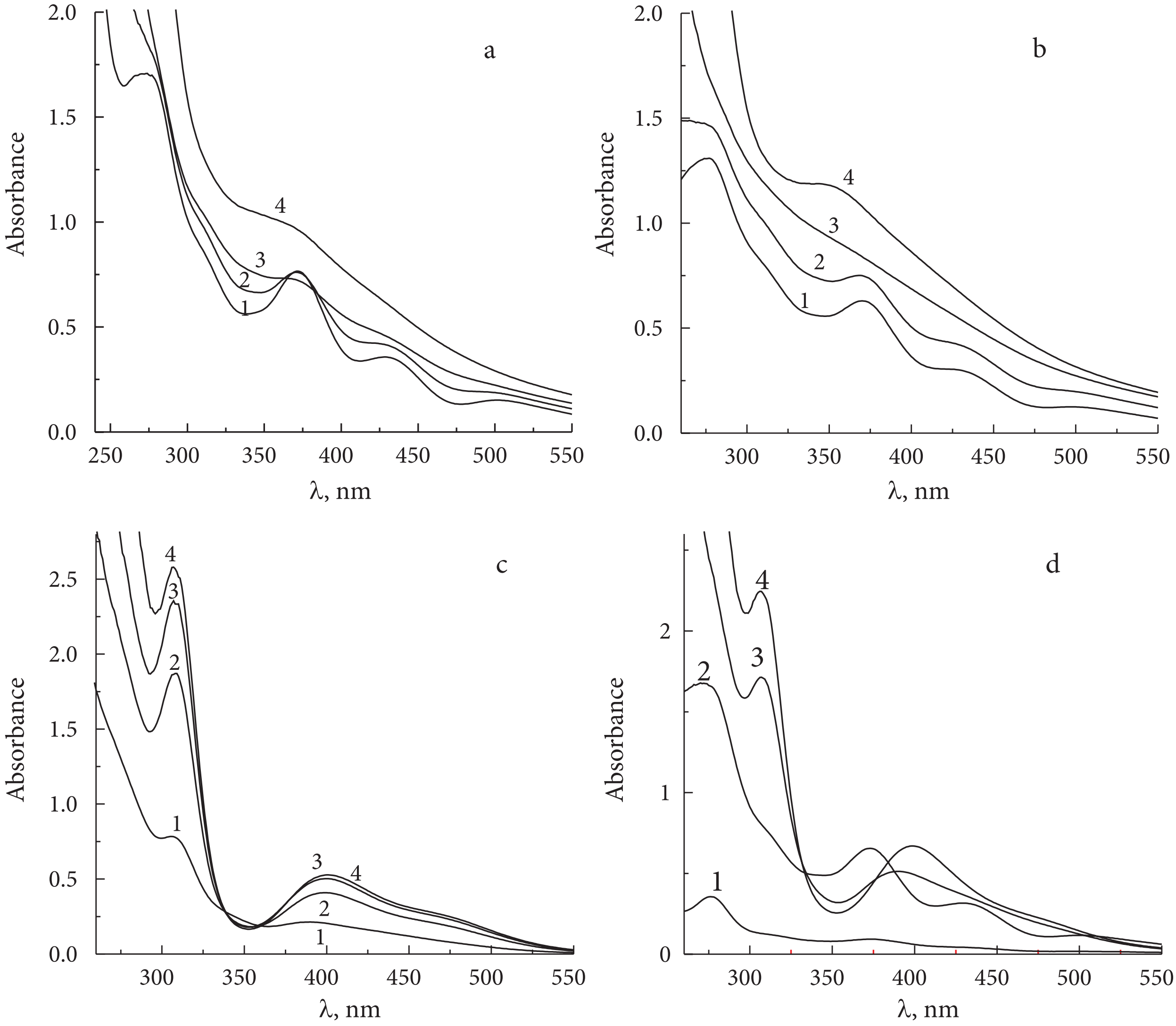

Fig. 3. Dependence of Pt–Sn complexes light absorption on Sn(II) concentration (mM): *1* – 5, *2* – 10, *3* – 20, *4* – 50; Pt(IV) – 0.3 mM, HCl: *a* and *b* – 0.5, *c* and *d* – 1.1 M; *a* and *c* – as prepared, *b* and *d* – after two days. Solutions diluted 1 : 4 with HCl 1 : 10

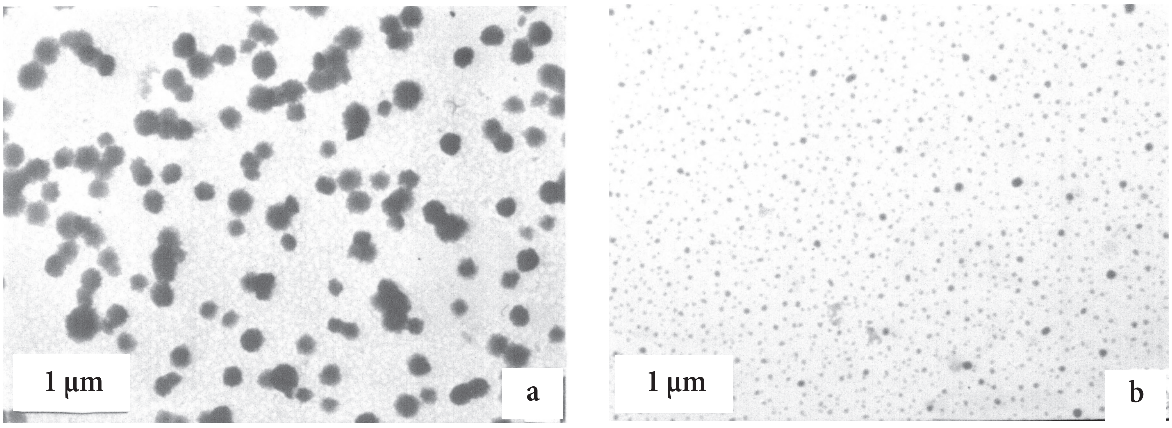

Fig. 4. TEM image of particles of Pt–Sn complexes prepared in solutions containing the initial concentrations: Pt(IV) – 0.3 mM, Sn(II) – 50 mM, HCl: *a* – 0.5, *b* – 1.0 M

formation of form I, stabilizes the complexes and prevents their precipitation.

The platinum present in all the three forms of complexes with tin, according to the binding energies 72.9–73.4 eV (Fig. 5), corresponds radically to Pt(II). The formation of complex I that needs an addition of increased quantities of $SnCl_2$ and HCl, may be described as follows:

$$H_2PtCl_6 + 6SnCl_2 + HCl \rightarrow H_3[Pt(SnCl_3)_5] + SnCl_4 \qquad (1)$$

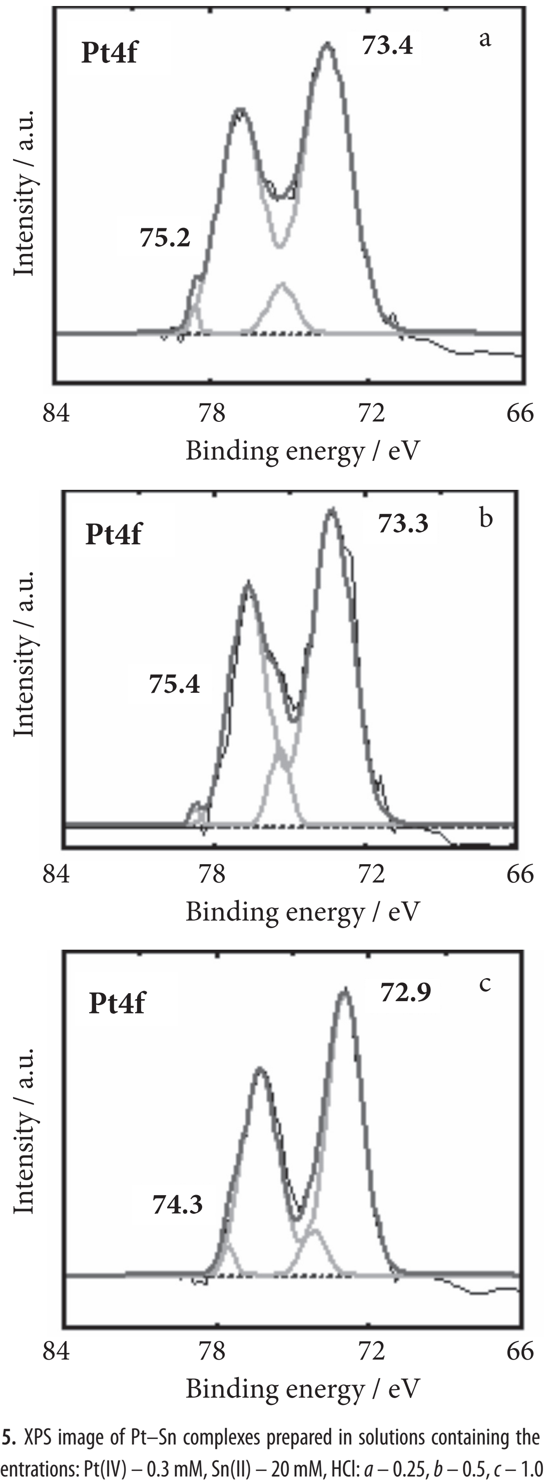

Fig. 5. XPS image of Pt–Sn complexes prepared in solutions containing the initial concentrations: Pt(IV) – 0.3 mM, Sn(II) – 20 mM, HCl: *a* – 0.25, *b* – 0.5, *c* – 1.0 M

because the binding energy of the Pt(II) ion in $(CH_3)_3[Pt(SnCl_3)_5]$ is very similar (73.3 eV) [19]. Complex II that is formed at lower concentration of $SnCl_2$ corresponds to the complex $(CH_3)_3Cl_2Pt(SnCl_3)_2]_2$ with the binding energy of platinum 73.4 eV, thus, its formation may be described by the reaction:

$$H_2PtCl_6 + 3SnCl_2 \rightarrow H_2[Cl_2Pt(SnCl_3)_2] + SnCl_4 \qquad (2)$$

According to the binding energy of platinum in complex III with Sn(II) (~73 eV), it conforms to Pt(II), and this complex may hardly be described by formula $[Pt_3Sn_8Cl_{20}]^{-4}$ with Pt(0). For the formation of complex III, a higher concentration of $SnCl_2$ is necessary, as compared to that for form II. Therefore, the reaction of its formation may be written as follows:

$$3H_2PtCl_6 + 11SnCl_2 \rightarrow H_2\,[Pt_3(SnCl_3)_8] + 3SnCl_4 + 4HCl \qquad (3)$$

The changes in the composition of platinum–tin complexes over time, and herewith in the light absorption spectra, are related to a decrease in Sn(II) concentration due to its fast oxidation by the air oxygen. The highest rate of Sn(II) oxidation is observed in the solutions with the highest HCl concentration (Fig. 6). The increase in HCl concentration accelerates Sn(II) oxidation in both cases, in the solutions with and without platinum, because an increase in HCl concentration promotes Sn(II) oxidation to Sn(IV):

$$SnCl_2 + 0.5O_2 + 2HCl \rightarrow SnCl_4 + H_2O \qquad (4)$$

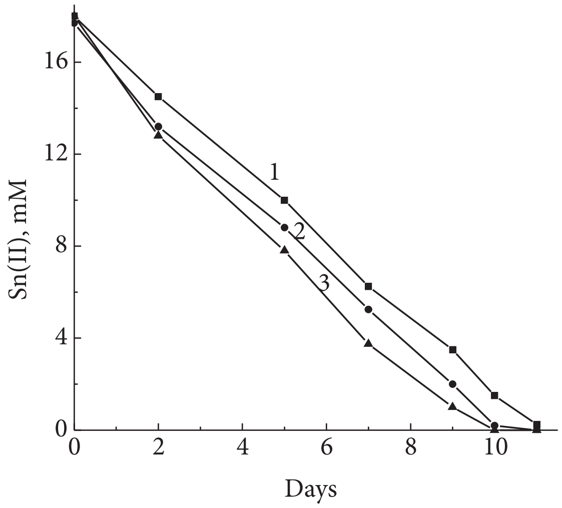

Fig. 6. Decrease in Sn(II) concentration over time. The initial concentration of Pt(IV) – 0.5 mM, HCl: *1* – 0.5, *2* – 0.7, *3* – 1.0 M

Sn(II) being coordinated with platinum in a complex (such as $SnCl_3^-$) is oxidized heavier than free Sn(II), therefore, an increase in the initial Pt(IV) concentration in solutions somewhat slows down the Sn(II) oxidation.

The complex with the initial HCl 1 M and $SnCl_2$ 50 mM concentrations changes over time (Fig. 7) showing gradual transition from complex I to II. This change may be described by the reaction:

$$H_3[Pt(SnCl_3)_5] \leftrightarrow H_2[Cl_2Pt(SnCl_3)_2] + 3SnCl_2 + HCl \qquad (5)$$

The oxidation of Sn(II) shifts this reaction to the right, so the whole reaction may be expressed as follows:

$$H_3[Pt(SnCl_3)_5] + 1.5O_2 + 5HCl \rightarrow$$
$$H_2[Cl_2Pt(SnCl_3)_2] + 3SnCl_4 + 3H_2O \qquad (6)$$

The transition of complex II to III is also related to Sn(II) oxidation:

$$3H_2[Cl_2Pt(SnCl_3)_2] + 3.5O_2 + 4SnCl_2 \rightarrow$$
$$H_2[Pt_3(SnCl_3)_8] + 2SnCl_4 + 7H_2O \qquad (7)$$

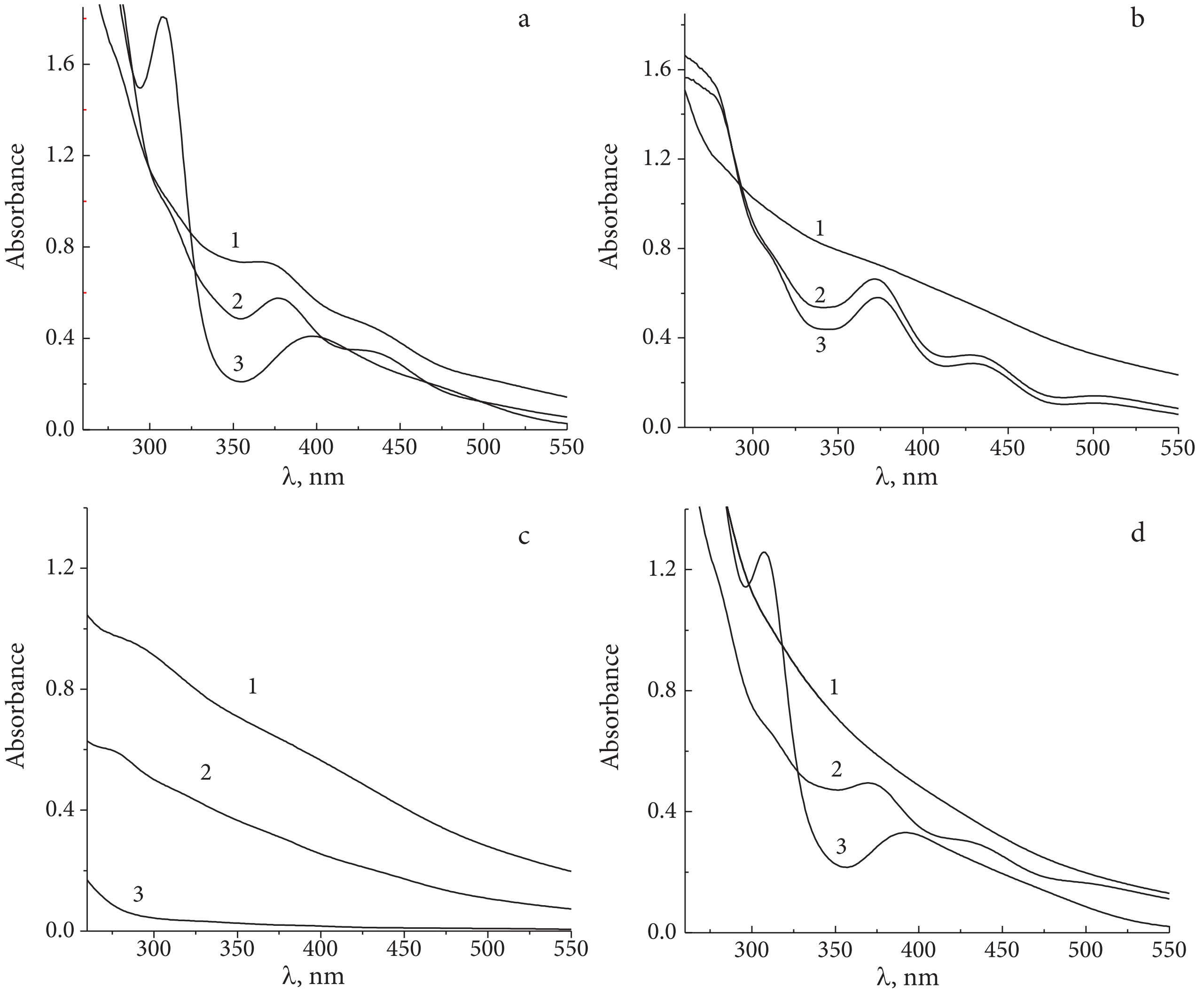

Fig. 7. Change of light absorption of the Pt–Sn complexes containing the initial concentrations: Pt(IV) – 0.5 mM, Sn(II) – 45 mM, HCl (M): 1 – 0.5, 2 – 0.7, 3 – 1.0; *a* – as prepared, *b* – after 5 days, *c* – after 11 days, *d* – after adjusting Sn(II) and HCl concentrations. Solutions diluted 1 : 4 with HCl 1 : 10

The $H_3[Pt(SnCl_3)_5]$ complex formed with the highest HCl concentration decomposes foremost (Fig. 7c). It discolors when the ratio of the residual Sn(II) to platinum in the solution becomes lower than 5. This fact confirms that platinum in these derivatives is bonded to Sn(II). With an addition of the missing amounts of $SnCl_2$ and HCl (as compared to the initial ones) in the solutions, the spectra of which are shown in Fig. 7c, all the complexes recover their initial colors and the spectra of the obtained solutions (Fig. 7d) become close to the initial ones.

The influence of the concentration of Cl^- ions from KCl to the formation of Pt(II)–Sn(II) complexes was investigated with the purpose to diminish the initial HCl concentration that increases the rate of Sn(II) oxidation and, therefore, decreases the time of the existence of complexes. The spectra of the compounds obtained in the solutions with the same initial Pt(IV), Sn(II) and HCl concentrations (with the exception of curve 4 obtained at HCl 1.3 M) are given in Fig. 8a. An addition of 0.6–1.3 M KCl leads to the formation of $H_3[Pt(SnCl_3)_5]$ complex, therefore, this form is obtained when the total concentration of Cl^- in the solution, not solely HCl, is over 1 M (curves 2, 3). An inconsiderable change over time in the spectrum of the complex obtained with the total Cl^- concentration 1.2 M is observed foremost: after 4 days, the former maximum at 400 nm shifts to 390 nm, showing gradual transition to $[Cl_2Pt(SnCl_3)_2]^{2-}$ complex at the time when the spectra of the solutions containing a higher total Cl^- concentration remains stable. However, the fastest oxidation of Sn(II) in the solution with the highest HCl concentration leads to a decomposition of the complex after 5 days (Fig. 8b). In this case, the complex disintegrates without passing to form II. The spectrum obtained shows that the following reaction is possible at higher HCl concentrations in the solution:

$$H_3[Pt(SnCl_3)_5] + 2.5O_2 + 9HCl \rightarrow H_2PtCl_4 + 5SnCl_4 + 5H_2O \qquad (8)$$

After the next three days, the analogous complex, formed with the total Cl^- concentration 1.8 M (0.5 M HCl and 1.3 M KCl), also disintegrates in the same manner, at the time when the identical initial complex, formed with a lower KCl concentration (0.7 M), passes to the $[Cl_2Pt(SnCl_3)_2]^{2-}$ complex.

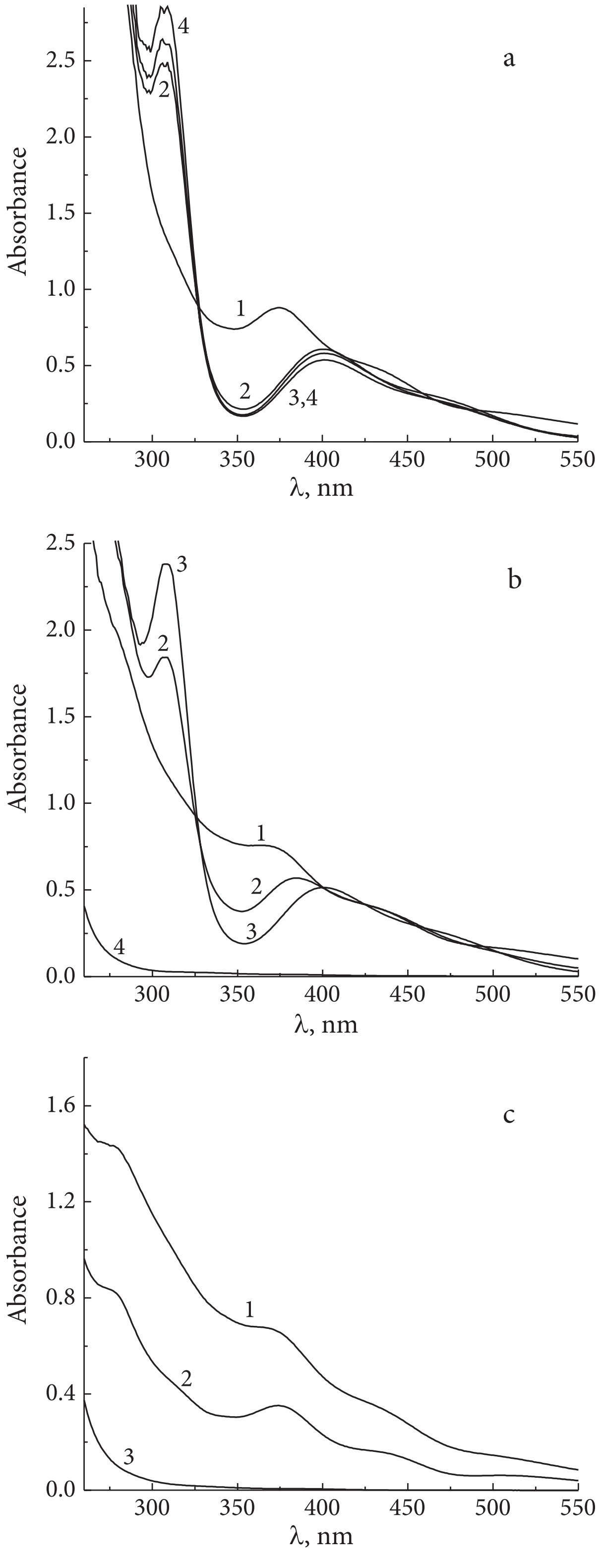

Fig. 8. Light absorption of Pt–Sn complexes formed in solutions containing the initial concentrations: Pt(IV) – 0.1, Sn(II) – 50 mM, HCl – 0.5 M. Additionally, *2* – contains 0.7, *3* – 1.3 M KCl, *4* – 1.2 M HCl; *a* – as prepared; *b* – after 5 days, *c* – after 8 days. Solutions diluted 1 : 4 with HCl 1 : 10

CONCLUSIONS

Three forms of Pt(II) complexes with Sn(II) may be formed subject to the initial $SnCl_2$, HCl and KCl concentrations in the reaction between Pt(IV) and Sn(II) solutions. An increase in $SnCl_2$ concentration stabilizes the complexes. The transition of $H_3[Pt(SnCl_3)_5]$ complex to $H_2[Cl_2Pt(SnCl_3)_2]$, and of $H_2[Cl_2Pt(SnCl_3)_2]$ complex to $H_2[Pt_3(SnCl_3)_8]$ observed over time is related to Sn(II) oxidation in the solutions by the air oxygen. An increase in temperature accelerates the transition. $H_3[Pt(SnCl_3)_5]$ complex turns to H_2PtCl_4 without a transition to form II in the solutions with higher HCl or total Cl^- concentrations.

A certain HCl : $SnCl_2$ ratio is necessary for the formation of rather stable complexes with platinum. An increase in HCl concentration accelerates Sn(II) oxidation and leads to the decay of the complexes over time. With an addition of the missing amounts of Sn(II) and HCl, platinum–tin complexes regained their initial state.

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Rima Tarozaitė, Aldona Jagminienė, Vitalija Jasulaitienė, Marija Kurtinaitienė

KOMPLEKSŲ, GAUNAMŲ REAGUOJANT ALAVUI(II) SU PLATINA(IV), SPEKTROMETRINIS TYRIMAS

Santrauka

Šviesos absorbcijos ir rentgeno fotoelektroninės spektroskopijos metodais nustatyta, kad, reaguojant Pt(IV) ir Sn(II) tirpalams, priklausomai nuo HCl, $SnCl_2$ ir bendros Cl^- koncentracijų dydžio susidaro trys Pt(II) ir Sn(II) kompleksų formos. Komplekso $[H_3Pt(SnCl_3)_5]$ perėjimas į $H_2[Cl_2Pt(SnCl_3)_2]$ ir komplekso $H_2[Cl_2Pt(SnCl_3)_2]$ perėjimas į $H_2[Pt_3(SnCl_3)_8]$, stebimas laikui bėgant, yra susijęs su Sn(II) oksidacija. Pakėlus temperatūrą šis perėjimas paspartėja. Pradinius kompleksus galima vėl atstatyti papildžius trūkstamus $SnCl_2$ ir HCl kiekius. Padidinus $SnCl_2$ koncentraciją stabilizuojami kompleksai.